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L2: Entry 2 of 4

File: USPT

Oct 25, 1988

DOCUMENT-IDENTIFIER: US 4780156 A

TITLE: Water resistant sensitizing additive for ammonium nitrate blasting agents

Brief Summary Text (57):

As indicated, the additive compositions of our invention can vary from light to very substantial water resistance, depending on the water thickener content and degree of cross-linking. More importantly, the energy and sensitivity additives impart the ability for water wet sections to detonate effectively in conjunction with gel formation. These additives also impart explosive strengths varying from that of typical ANFO to the increased strength of metalized ANFO blasting agents, depending on the additive. Detonation velocities of our additive and ANFO compositions are generally faster than ANFO alone, when using the same ammonium nitrate prill, granulation, bore hole diameter and confinement. Because water content of ANFO mixtures with our additive is accidental, depending on the concentration and rate of flow of water, the density may vary locally in a drill hole from 0.80 to 1.25 gms/cc. Water resistant blasting agents formulated from compositions of our invention may contain 45 to 95 percent ammonium nitrate prills, 1.5 to 9 percent liquid organic fuels, 0 to 40 percent sodium nitrate, calcium nitrate or a combination of both, from 1 to 10 percent water thickener, 0.005 to 1 percent of cross-linker, 1 to 20 percent energy and sensitivity additives, 0 to 5 percent solid carbonaceous fuels, and 0 to 5 percent inert solids. Water content supplied by the drill hole or other environmental conditions may vary accidentally from 0 to 30 percent in actual use. Preferred compositions of our invention contain from 60 to 90 percent ammonium nitrate, 2.5 to 6 percent liquid organic fuels, 0 to 20 percent sodium nitrate, calcium nitrate or both, 1 to 5 percent guar gum flour, 0.005 to 0.5 percent cross-linker, 2.5 to 12 percent energy and sensitivity additives, 0.8 to 3 percent solid carbonaceous fuels and 0.3 to 1.5 percent inert solids. Oxygen balance of the preferred compositions is between +5.0 and -18.0, with formulations for underground use being oxygen balanced.

Current US Class (1):149

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L3: Entry 3 of 3

File: USPT

Feb 25, 1986

DOCUMENT-IDENTIFIER: US 4572075 A

**** See image for Certificate of Correction ****

TITLE: Methods and apparatus for loading a borehole with explosives

Brief Summary Text (19):

To increase the lower weight strength of traditional slurry explosives, some commercial formulators have added up to 25% ammonium nitrate prills to the slurry in order to reduce the effective water content. Others have developed formulations where granular aluminum, a very expensive ingredient (which can cost five to eight times more per pound than the base slurry), is added in quantities up to 25% to increase the weight strength of the explosive.

Current US Class (1):102

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L6: Entry 2 of 13

File: USPT

Oct 26, 1999

DOCUMENT-IDENTIFIER: US 5972137 A

TITLE: Explosives

Brief Summary Text (1):

THIS INVENTION relates to an explosive. More particularly, the invention relates to a method of sensitizing a base emulsion of the water-in-oil type to provide an emulsion explosive; and to an emulsion explosive when sensitized by the method.

Brief Summary Text (4):

By essentially organic is meant that the solvent contains no more than 50% by mass water, preferably no more than 25% and more preferably no more than 12%. By keeping water content at low levels, any undesirable effects of water, relating to reduced sensitivity or energy production upon detonation, can be resisted.

Detailed Description Text (11):

In a control test using the same proportions of constituents but using the same mass of water as the solvent for the sodium nitrite in the sensitizing solution instead of the ethylene glycol/water mixture containing dissolved sodium perchlorate, initiation of the sensitized emulsion could not be obtained in the same diameter cartridge with the same detonator, or even with higher strength initiators such as ANSTART (1.7 g PETN).

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L6: Entry 3 of 13

File: USPT

Oct 22, 1996

DOCUMENT-IDENTIFIER: US 5567911 A

TITLE: Particulate explosive, manufacturing method and use

Brief Summary Text (20):

As indicated, the solidified phases may be categorized into two general types, although intermediates may form and no sharp distinction can be found therebetween. In a first type the droplets are believed to solidify into an amorphous state without significant crystallisation. This type of emulsion can be obtained by methods known in the art and generally designated melt-in-fuels. Ordinarily a low water content, say below 5 percent by weight of the phase composition and preferably below 4 percent, is needed. Normally additional salt types are included in the composition in order e.g. to obtain a sufficiently low melting temperature. Melting temperatures above about 90 degrees centigrades are common. The amorphous solidification generally gives stable emulsion with suitable rigidity at lower levels of hard components in the fuel phase.

Brief Summary Text (21):

In a second type the salts in the discontinuous phase solidifies under at least partial crystallization. It is believed that in most instances several or multiple crystals are formed in each droplet. This crystalline solidification is generally preferred over the amorphous for best rheology and compaction properties. Crystallisation can be induced in salt compositions of abovesaid low water contents, e.g. by recrystallisation of the amorphous phase or by controlled release of crystallisation during cooling. It is preferred, however, to use oxidizer phase compositions of higher water content, which facilitates crystallisation and give final discontinuous phase composition mixtures of crystals together with saturated aqueous salt solution wherein the elementary crystals are believed to be clearly smaller than the phase droplets. Suitable water contents for these purposes are above 7 percent and preferably above 9 percent by weight of the phase composition. Too high water contents again may counteract crystallisation and the content should be below 20 percent and preferably below 16 percent. In both low and high water content compositions crystallisation can be initiated by by known means, e.g. U.S. Pat. No. 4,632,714, incorporated herein by reference, or preferably by the also known method of subjecting an already cooled emulsion containing supersaturated solution to sufficient friction or impact to activate crystallisation.

Brief Summary Text (25):

Also in similarity with common emulsion explosives the emulsion matrix for present purposes may include sensitizing agents, such as self-explosive additives but preferably density reducing agents. The requirement for such additives may vary strongly depending on the intended product use. The granulated product can be loosely filled into a bore-hole with substantial volumes of air between the granules. Charging under compaction may entrap varying amounts of air in the charge, thereby reducing the density reduction requirements for the matrix itself. Yet, in order to secure a reliable initiability of the matrix independent of charging conditions, it is preferred to include at least a minimum amount of density reducing agents in the emulsion, e.g. to a density below 1.25 g/cc or preferably below 1.2. Generally the density is kept above 0.8 and preferably also above 0.9 g/cc. Further density reduction may be used to obtain compositions of reduced strength although it is preferred to use other methods for this purpose as

will be further discussed below. Any known density reduction method can be used, such as air inclusion or chemical gassing although it is preferred to include microspheres such as thermoplastic spheres and in particular the more volume stable glass or mineral spheres.

Brief Summary Text (30):

Another preferred particulate composition is between the present emulsion containing granules and an inert and/or density reducing filler in order to give an overall composition of reduced energy content, e.g. for careful blasting. Any known kind of particulate filler or bulking agent can be used. Substantially homogeneous materials of high density can be exploited to provide for high composition density in spite of low strength, e.g. for the purpose of expelling water from drill holes. For this purpose inorganic materials are preferred, such as minerals or inert salts of the sodium chloride type, which latter type also may serve the purpose of reducing the igniting properties of the explosive. High density additives gives low segregation problems in the combined bulk material. To lower the overall density of the composition it is suitable to employ bulking agents of clearly lower density than that of the emulsion granules, e.g. below 0.8 g/cc. Advantageously the density is also lower than about 0.5 g/cc and more suitably lower than 0.3 g/cc. Porous inorganic bulking agents are substantially inept and can be used in the present compositions. Typical representatives for this filler category are expanded glasses, perlite, vermiculite, pumicite etc. The low filler mass introduced by lightweight materials permits use of organic materials with a certain fuel value. Organic fillers are available in bulk densities below 0.1 g/cc or even below 0.05 g/cc. Typical products of this kind suitable for the present purposes are expanded polymers of for example vinyl chloride, ethylene, phenol, urethane and especially styrene. Irregular particles, formed for example in subdivision of porous bulk materials, can be used although uniform particles and especially spherical particles, for example produced by expansion of discrete particles or droplets, are preferred. Satisfactory results have been obtained by spherical porous particles of preexpanded polystyrene foam beads. Especially for smaller addition amount the particle size is not critical and fine material of e.g. less than 1/10 or even 1/100 of granule size can be used. It is generally preferred though, especially for larger amounts, to use fairly large particle sizes and narrow size distributions. Particle sizes between 0.5 and 10 mm, or better between 1 and 5 mm, are then suitable. The bulking agent shall be added in an amount sufficient to reduce composition volume strength below the volume strength of the straight emulsion granules, here used as standard for relative volume strength. To be useful for careful blasting, the relative volume strength should be clearly lower than 100%, say below 80%, better below 60% and preferably also below 40%, established by calculations or experiments for specific compositions.

Brief Summary Text (35):

The second, solidification, step may be different for different oxidizer phase compositions. Low water content compositions intended to be solidified into amorphous form often requires nothing else than a fairly rapid cooling of the emulsion and absence of conditions facilitating crystallization. Once obtained in amorphous form, the state may be stable with low tendency for rearrangement. As well known in the art, cooling of emulsions and high water content emulsions in particular normally result in a supercooled state in which each droplet remains in solution despite its potential crystallization ability. This property is utilized and beneficial in normal emulsion explosive application but need to be overcome for the present purposes. Crystallization can be initiated in the emulsion during cooling, for example by securing presence of conditions facilitating crystallization, such as providing nucleating agents in accordance with known methods, by slow cooling or by disturbed cooling. It is generally preferred, however, to separate these actions and in a first step supercool the emulsion below its crystallization temperature, preferably to substantially ambient temperature in a conventional manner, and in a second step initiate crystallization. This method has proven to give emulsions of suitable rheological properties and also give the

advantage of full control over the crystallization moment, at any time between emulsion matrix formation and the charging operation. This freedom can for example be used to initiate crystallization in connection with or at the actual charging operation to thereby utilize the hardening and phase transition for better bore-hole charge cohesion. But it is generally preferred to initiate crystallization earlier to take full advantage of abovesaid benefits in manufacture, storage, transport and use. Initiation can take place between formation of the supercooled emulsion matrix and granulation but preferably it is made at or soon after granulation for reasons to be explained below. Second step initiation after cooling can be made with the same means mentioned for initiation during cooling but an additional and preferred possibility is to utilize the per se known method of releasing crystallization through mechanical stress, e.g. by sufficient friction, shear or impact to activate crystallization, which manifests itself through a distinct sensible energy release and temperature raise. The method gives a beneficial fine-grained crystal structure, which may be further amplified with optional addition in the oxidizer phase of crystal habit modifiers, such as formamide or urea.

Brief Summary Text (43):

The product may be used in any blasting application but the most typical applications are similar to those where ANFO is presently used although the water resistance of the present charges extends the use also to water filled holes. Special advantages are obtainable in careful blasting applications since the granules are easily combined with energy reducing fillers as described. The proposed compositions may then be used whenever a blasting composition with a volume strength reduced in relation to the compacted or uncompacted product is needed or whenever a blasting composition with readily variable strength is desired. Typical applications are contour blasting or pre-splitting above or underground as well as bench blasting for particular purposes. In underground mining and stoping, drift holes or production holes may be charged to full strength and the contour holes with reduced compositions. The reduced compositions may be plant-mixed but greater flexibility may be achieved by on-site mixing of the present granules with the energy reducing filler.

CLAIMS:

4. The explosive of claim 1, wherein the emulsion has a water content above 5 percent by weight.

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L6: Entry 5 of 13

File: USPT

Dec 5, 1995

DOCUMENT-IDENTIFIER: US 5472529 A

**** See image for Certificate of Correction ****

TITLE: Explosive composition and method for producing the same

Brief Summary Text (4):

Slurry explosives and emulsion explosives are typical of the conventional hydrous explosives. In these explosives, the active explosive components comprising an oxidizer solution, an inflammable material and a sensitizer and the bubbles are held stably in high concentrations in a mass in the presence of a sizing agent, and these explosives are usually detonated by means of a detonator. In the slurry explosives, the aerated bubbles or chemical bubbles are usually allowed to exist in the explosive composition to let them play a role like a sensitizer, and guar gum is used as sizing agent to compose an aqueous gel. In the emulsion explosives, an oxidizer solution and an oil serving as an inflammable agent are combined to form a W/O type emulsion in the presence of a surfactant serving as a sizing agent. The bubbles in these explosives comprise glass or resinous microballoons, besides the aerated bubbles.

Detailed Description Text (6):

The water content in the composition of this invention is usually in the range of 3 to 20% by weight based on the whole composition. When the water content is below this range, the solid content of the explosive composition increases to affect the stable blasting performance thereof, while a too high water content results in a reduced detonating performance.

Detailed Description Text (8):

The organic hollow microspheres used in the composition of the present invention are not specifically defined; they may be hollow spheres containing a gas or air in the inside hollow portion or hollow bodies having closed or open spaces therein, but hollow spherical bodies are preferred for efficiently forming the hot spots where the explosive charge is detonated. The gas held in the organic hollow spheres may be air, a low-boiling point hydrocarbon, other inflammable gas, or a mixture thereof. The recommendable particle size of the organic hollow microspheres is about 1,000 .mu.m or less in diameter. When the particle size exceeds this limit, the hot spots for initiating explosion are reduced in number, making it difficult to produce good detonation property. More preferably the organic hollow microspheres have a particle diameter of 20 to 200 .mu.m as these spheres can provide a stabilized explosion without lowering the velocity of detonation. The film thickness of the organic hollow microspheres is not critical; it may be properly selected as far as the film has enough strength to give a space for accommodating the explosive composition. Usually, the film thickness is 0.1 to 5 .mu.m. In case the organic high-molecular weight compound forming the organic hollow microspheres is the thermoplastic type, there are used those microspheres whose film thickness in the foamed state is about 0.1 to 2 .mu.m since they are required to be capable of being foamed by heating in the explosive composition. The organic hollow microspheres in the explosive composition of this invention are usually of a bulk density of about 0.01 to 0.3 as measured in a dry state. The amount of the organic hollow microspheres in the composition is usually about 2 to 15% by weight based on the whole composition. The density of the explosive composition can be controlled by the amount of the organic hollow microspheres.

Generally, when the ratio of the organic hollow microspheres in the composition is too low, the detonating efficiency is lowered, and it also becomes difficult to maintain a stabilized blasting performance for a long time. On the other hand, when the ratio of said organic hollow microspheres is too high, the power of explosion is lowered to jeopardize the blasting reliability.

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L6: Entry 6 of 13

File: USPT

Feb 19, 1991

DOCUMENT-IDENTIFIER: US 4994124 A

TITLE: Sensitized explosive

Brief Summary Text (2):

A wide range of explosives for commercial blasting are currently available in a variety of densities, strengths, sensitivities, physical forms and prices. Recent developments in the production and use of ammonium nitrate-fuel oil (ANFO) emulsion and slurry explosives have, to a large degree, resulted in the replacement of more traditional explosives. However, still in current use in the industry, are the conventional "stick-type" explosives, containing nitroglycerin (NG) or ethylene glycol dinitrate (EGD) as sensitizers. The stick-type explosives maintain their commercial utility because of their high strength, reliability, sensitivity, and competitive cost.

Brief Summary Text (8):

All of the emulsions described in the patents referred to hereinabove comprise less than 5% water. This limitation results in increased difficulty in the preparation of the dry explosive in that either a melt processing technique is used whereby dry ammonium nitrate is emulsified into a molten TNT mixture, or, a low water emulsion is prepared. Both the "melt" and the low water emulsion compositions have been found to provide suitably sensitive materials for blasting, and as sensitizers. However, these materials must be manufactured at high temperatures in order to melt the oxidizer salt or to make a low water content aqueous solution. At these high temperatures, the emulsion explosive mixture is increasingly more shock sensitive. Further, after production, the dry sensitized explosives are sensitive to friction and low impact which makes processing the final dry product difficult and/or hazardous.

Brief Summary Text (10):

It is an object of the present invention to provide a high strength explosive.

Brief Summary Text (22):

The water content of the oxidizer phase of the particulate explosive, after crystallization, is 5 to 30% by weight of the total weight of the composition. Preferably, the water content is between 8 and 15% by weight, and more preferably between 10 and 12% by weight, in order to ensure that the processing temperature is below 95.degree. C. and to ensure a good sensitivity in the resulting crystallized salt.

Brief Summary Text (25):

Control of the sensitivity of the composition by the use of microballoons, for example, also allows the same emulsion to be used to prepare a cast explosive, wherein the emulsion is poured into a mold and allowed to crystallize in the shape of the mold.

Detailed Description Text (68):

Compositions 29 to 32 show cast explosives that have been sensitized by TNT or sodium perchlorate and thus are sensitized by internal and external sensitizers. Composition 30 illustrates a high water content cast explosive.

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L6: Entry 8 of 13

File: USPT

Jul 4, 1989

DOCUMENT-IDENTIFIER: US 4844756 A

TITLE: Water-in-oil emulsions

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A problem with water-in-oil hydraulic fluids is that they have a tendency to cause wear on metallic pump parts and other equipment with which they come into contact. The water phase, though dispersed in the oil phase, creates wear problems that are not encountered with straight petroleum oil compositions. Another problem is that the water phase tends to corrode the metallic parts it contacts. Water-phase additives which have been employed previously to reduce wear and/or corrosion have had the disadvantage that they tend to precipitate from the emulsion, particularly when the water content becomes reduced during use. The omission of water-phase additives, on the other hand, is undesirable since it is frequently impossible to obtain satisfactory wear- and/or corrosion-resistance by the use of additives which are dissolved only in the oil phase.

Brief Summary Text (6):

Explosive emulsions are typically constituted of continuous organic fuel or oil phase in which discrete droplets of an aqueous solution of an oxygen-supplying source are dispersed as a discontinuous phase. Such compositions are conventionally described as water-in-oil explosive emulsion compositions, and examples thereof have been described, inter alia, in U.S. Pat. Nos. 3,447,978; 3,985,593; 4,008,110; 4,097,316; 4,104,092; 4,110,134; 4,149,916; 4,149,917; 4,218,272; 4,259,977; 4,357,184; 4,371,408; 4,404,050; 4,409,044; 4,453,989; and 4,534,809; and European Application Publication No. 0,155,800 A1, which are incorporated herein by reference. Formation of these explosive emulsions is generally effected in the presence of an emulsifier which is selected to promote subdivision of the droplets of the oxidizer phase and dispersion thereof in the continuous phase. While many of the emulsifiers described in the prior art are meritorious, none have provided emulsion stability characteristics that are entirely satisfactory. Additionally, with most emulsifiers used in the prior art, selection of the fuel or oil for the continuous phase is generally limited to highly-refined, highly paraffinic oils such as white oils.

Brief Summary Text (27):

In still another preferred embodiment, the invention provides for an explosive emulsion comprising:

Brief Summary Text (48):

These emulsions can also be used as explosive emulsions. For such explosive emulsions the functional additive (D) is preferably an oxygen-supplying salt. In such explosive emulsions the oil phase (A) is preferably present at a level in the range of from about 2% to about 15% by weight, more preferably from about 4% to about 8% by weight based on the total weight of said emulsion. The aqueous phase (B) is preferably present at a level in the range of from about 85% to about 98% by weight, more preferably from about 92% to about 96% by weight based on the total weight of said emulsion. Component (C) is preferably present at a level in the range of from about 4% to about 40% by weight, more preferably from about 12% to about 20% by weight based on the total weight of the oil phase (A). The functional additive (D) is preferably present at a level in the range of about from 70% to

about 95% by weight, more preferably from about 85% to about 92% by weight, more preferably from about 87% to about 90% by weight based on the total weight of the aqueous phase (D).

Detailed Description Text (62):

Explosive Emulsions:

Detailed Description Text (63):

When the inventive emulsions are employed as explosive emulsions, such emulsions typically contain other additional additives such as sensitizing components, oxygen-supplying salts, particulate light metals, particulate solid explosives, soluble and partly soluble self-explosives, explosive oils and the like for purposes of augmenting the strength and sensitivity or decreasing the cost of the emulsion.

Detailed Description Text (64):

The sensitizing components are distributed substantially homogeneously throughout the emulsions. These sensitizing components are preferably occluded gas bubbles which may be introduced in the form of glass or resin microspheres or other gas-containing particulate materials. Alternatively, gas bubbles may be generated in-situ by adding to the composition and distributing therein a gas-generating material such as, for example, an aqueous solution of sodium nitrite. Other suitable sensitizing components which may be employed alone or in addition to the occluded or in-situ generated gas bubbles include insoluble particulate solid self-explosives such as, for example, grained or flaked TNT, DNT, RDX and the like and water-soluble and/or hydrocarbon-soluble organic sensitizers such as, for example, amine nitrates, alkanolamine nitrates, hydroxyalkyl nitrates, and the like. The explosive emulsions of the present invention may be formulated for a wide range of applications. Any combination of sensitizing components may be selected in order to provide an explosive composition of virtually any desired density, weight-strength or critical diameter.

Detailed Description Text (65):

The quantity of solid self-explosive ingredients and of water-soluble and/or hydrocarbon-soluble organic sensitizers may comprise up to about 40% by weight of the total emulsion. The volume of the occluded gas component may comprise up to about 50% of the volume of the total explosive emulsion.

Detailed Description Text (66):

Optional additional materials may be incorporated in the explosive emulsions of the invention in order to further improve sensitivity, density, strength, rheology and cost of the final explosive. Typical of materials found useful as optional additives include, for example, emulsion promotion agents such as highly chlorinated paraffinic hydrocarbons, particulate oxygen-supplying salts such as prilled ammonium nitrate, calcium nitrate, perchlorates, and the like, particulate metal fuels such as aluminum, silicon and the like, particulate non-metal fuels such as sulfur, gilsonite and the like, particulate inert materials such as sodium chloride, barium sulphate and the like, water phase or hydrocarbon phase thickeners such as guar gum, polyacrylamide, carboxymethyl or ethyl cellulose, biopolymers, starches, elastomeric materials, and the like, crosslinkers for the thickeners such as potassium pyroantimonate and the like, buffers or pH controllers such as sodium borate, zinc nitrate and the like, crystals habit modifiers such as alkyl naphthalene sodium sulphonate and the like, liquid phase extenders such as formamide, ethylene glycol and the like and bulking agents and additives of common use in the explosives art.

Detailed Description Text (68):

A preferred method for making the water-in-oil explosive emulsions of the invention comprise the steps of (1) mixing the water, inorganic oxidizer salts and, in certain cases, some of the optional water-soluble compounds, in a first premix, (2)

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L6: Entry 9 of 13

File: USPT

Oct 4, 1988

DOCUMENT-IDENTIFIER: US 4775431 A

TITLE: Macroemulsion for preparing high density explosive compositions

Brief Summary Text (6):

Various attempts have been made to increase the density or bulk strength of ANFO, and thereby provide more energy per volume. Some examples of these attempts include the use of high density additive fuels (e.g., ferrophosphorous), crushing the ammonium nitrate, and using thickened water-based AN slurries. These attempts have met with limited success. The use of high density fuels require special equipment for addition of the fuels to the prills which increases the cost of the explosive. Similarly, special equipment and extra personnel are required for partially crushing the prills which also leads to increased costs. Slurries have the problem of lacking sensitivity and require the addition of sensitizing agents as well as the use of additional equipment.

Brief Summary Text (9):

Numerous other patents also describe explosive emulsions that incorporate oxidizing agents in the aqueous phase of the emulsion. U.S. Pat. No. 3,161,551 to Egly, et al. discloses a water-in-oil emulsion containing 50-70% by weight of ammonium nitrate, 15-35% water, 5-20% of an organic sensitizer and a small amount of emulsifiers. Egly teaches to combine this AN-based emulsion with particulate ammonium nitrate so as to fill all the spaces between the particles. U.S. Pat. No. 3,356,547 to Berthmann, et al. describes an emulsion containing nitroglycerine that is used in combination with solid AN particles.

Brief Summary Text (18):

The present invention provides an explosive composition that can be used as a replacement for ANFO, or in combination with ANFO, while using the same equipment and essentially the same processes as are currently used for ANFO and providing an explosive composition with a greater density and bulk strength than ANFO. These features result in a very desirable explosive composition.

Detailed Description Text (5):

These examples illustrate that the higher the water content in the emulsion the greater the density rise. Even with only 10 grams of water, a significant increase occurs in the density of the explosive composition over ANFO within one hour. As the amount of water in the macroemulsion is increased, the density of the explosive composition increases even further. Similarly, as the amount of macroemulsion is increased relative to the solid oxidizer, the density of the resulting explosive composition also increases.

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L6: Entry 11 of 13

File: USPT

Sep 15, 1987

DOCUMENT-IDENTIFIER: US 4693765 A

TITLE: Gel type slurry explosive and matrix and method for making same

Brief Summary Text (6):

Over the years many attempts have been made to increase the explosive power of a given amount of ANFO, to make it water resistant so that it can be used in damp and water immersed environments such as in blast holes containing water, and to make it easier to handle and load into bore holes for blasting. A common way of increasing the explosive strength of the ANFO is by the addition of a metal powder such as aluminum powder. A way of making the explosive easier to handle in the filling of bore holes is to make the explosive in the form of a flowable slurry, and slurry explosives have become very popular. By thickening the slurry in various ways, a slurry can be made to have varying degrees of water resistance.

Brief Summary Text (7):

The term strength of an explosive refers to the energy content of the explosive, which in turn, is a measure of force or power it can develop and its ability to do work. The term strength is used with ammonium nitrate based explosives to compare their energies to ANFO and is measured in kilo-calories per gram. In the case of ANFO, which is the base of reference, the value of weight strength is 1.0 k-cal/gm. The other term, bulk strength, compares explosives on a bulk or volume basis, and is a function of the density of the material. The higher the density, the higher the bulk strength. Its unit of measure is kilo-calories per cubic centimeter, abbreviated k-cal/cc.

Brief Summary Text (8):

The most common form of slurry explosives are emulsions which are mixed with ANFO. The emulsions are oil in water or water in oil emulsions which contain oxidizers and fuels in various proportions and are mixed with ANFO wherein the emulsion generally makes up 30% to 50% of the final mixture. Emulsion explosives generally have less weight strength than ANFO, but usually have increased density.

Brief Summary Text (12):

There remains a need for an inexpensive, easily made slurry explosive having increased explosive strength over ANFO and which is highly water resistant.

Brief Summary Text (14):

According to the invention, an economical slurry explosive having good water resistant properties, and generally increased explosive strength over ANFO is easily made by a matrix material with ANFO wherein the matrix material makes up only between about 14% to about 40% of the mixture. The mixture forms a water gel and may be cross-linked to increase water resistance.

Brief Summary Text (15):

The matrix material includes an oxidizer solution which also acts as a sensitizer and is selected from the group consisting of an aqueous sodium perchlorate solution, an aqueous calcium nitrate solution, an aqueous ammonium perchlorate solution, or a combination of such solutions, a liquid fuel such as ethylene glycol or fuel oil, and a thickening agent such as a guar gum in combination with an acid, such as glacial acetic acid, which also acts to adjust the pH of matrix. The

sodium perchlorate solution is preferably a 55% by weight aqueous solution and may comprise up to about 97% of the matrix. Since the sodium perchlorate solution makes up the bulk of the matrix and is relatively expensive, the cost of the matrix may be reduced, along with some of the increased explosive strength, while maintaining the desirable slurry and water resistant properties of the finished explosive, by substituting a calcium nitrate solution for all or part of the sodium perchlorate solution. The calcium nitrate solution is preferably about a 50% aqueous solution. The matrix takes the form of viscous, honey-like material. The ammonium perchlorate solution currently is much more expensive than the sodium perchlorate solution so is currently not preferred for use in the invention, but if used would be aqueous solution similar to the sodium perchlorate solution.

Detailed Description Text (11):

With the matrix formulation shown above, the explosive power of the finished slurry is increased from the 1.0 k-cal/gm weight strength of ANFO to about 1.16 to 1.20 k-cal/gm weight strength. This compares with a decrease in explosive power when emulsions are used to mix with ANFO.

Detailed Description Text (17):

The second matrix produces a less sensitive explosive with a critical diameter of about five inches. Thus, the bags or cartridges into which the explosive is loaded must have diameters at least five inches. Also, the slurry has less explosive strength than that made with the first matrix, and in terms of weight strength, is less than the weight strength for ANFO. However, because of its density, its bulk strength is just slightly higher than that of ANFO and because it is a gel, it exhibits excellent water resistant properties. The cost of the explosives made according to the above formula is less than the cost of ANFO.

Detailed Description Text (23):

This third matrix formulation produces an explosive with about the same sensitivity as the second embodiment with a critical diameter of about five inches, but produces an explosive having significantly increased weight strength over the second embodiment, i.e. a weight strength of about 1.1 k cal/gm, which is just over that of ANFO. Because of its density, however, its bulk strength is significantly higher than that of ANFO, and the cost is just slightly more than the cost of ANFO.

CLAIMS:

23. A matrix composition according to claim 1, wherein the water content of the matrix is between about 42% and 49%.

[First Hit](#) [Fwd Refs](#)

Generate Collection

Print

L6: Entry 12 of 13

File: USPT

Nov 26, 1985

DOCUMENT-IDENTIFIER: US 4555278 A

TITLE: Stable nitrate/emulsion explosives and emulsion for use thereinBrief Summary Text (11):

Binet et al.'s consideration of storage stability is directed for the most part at the explosive emulsion itself. The patentees mention that all known prior art water-in-soil emulsions suffer from lack of stability owing to the seeding effect. Binet et al. also imply that the seeding effect is a problem in AN-doped emulsions, although they do not explain how this can be so in microemulsions containing relatively large AN particles. Moreover, Binet et al. require an expensive polymeric emulsifier, and an optional emulsion stabilizer, to achieve improved stability in their microemulsion.

Brief Summary Text (21):

In a further embodiment, the present invention provides a water-in-oil emulsion adapted to be blended with inorganic nitrate prills by one embodiment of the method of the invention to form a stable explosive, said emulsion comprising

Brief Summary Text (27):

The amount of inorganic oxidizing salt (the oxidizer) present in the "high oil" emulsion of the invention is insufficient for the complete combustion of the fuel therein, as is evidenced by the emulsion's negative oxygen balance. This oxidizer-deficient emulsion is converted into a product having a more positive oxygen balance and satisfactory explosive properties by blending with fuel-deficient or, preferably, substantially fuel-free inorganic nitrate, preferably AN, prills. By virtue of its relatively low viscosity, the oxidizer-deficient emulsion can be blended with these prills with low shear so as to produce a preferred explosive emulsion/nitrate blend of the invention containing about from 20 to 70 percent by weight of prills and a sensitizing amount of dispersed gas bubbles or voids, the blend being essentially oxygen-balanced, i.e., having an oxygen balance more positive than about -25 percent, and preferably in the range of about from -10 to +5 percent. Blends made from the preferred in situ emulsion and about from 20 to 50 percent prills have a viscosity in the range of about from 2500 to 20,000 poise, a viscosity in this range being maintainable for a period of several days.

Detailed Description Text (2):

The present invention is based on the discovery that the transport of water from the dispersed aqueous phase of the emulsion to the nitrate particles that are intermixed with the emulsion in nitrate/emulsion blends plays a major role in the instability of these blends, leading to a deterioration of product performance. This transfer of water results in an increase in the water content of the particulate nitrate, perhaps to a level of about 5 to 10 percent, and an increase in the salt concentration in the dispersed aqueous phase, approaching the saturation limit and the possibility that the salt may crystallize out. These combined effects can cause the structure of the emulsion/nitrate blend to deteriorate rapidly.

Detailed Description Text (7):

In a further preferred embodiment of the invention, the required structure formed by the inorganic nitrate particles and the components of the emulsion is provided

by controlling the cell size of the emulsion's internal phase (the aqueous salt solution droplets) so as to decrease the chemical driving force, i.e., the difference between the chemical potential of the water in the dispersed aqueous salt solution of the emulsion and the inorganic nitrate particles. A reduced chemical driving force minimizes the rate of water transport from the aqueous emulsion phase to the nitrate particles. The chemical potential of the components in the dispersed aqueous phase increases in inverse proportion to the radius of curvature of the cell (droplet). Therefore, smaller cell size increases the chemical potential of the water in the discontinuous phase, thereby increasing the driving force for water transport to the solid oxidizer. In the past, a smaller cell size (higher viscosity) has been recommended to increase the stability of emulsion explosives per se. For example, Clay (U.S. Pat. No. 4,181,546) recommends "a good shearing mixing" as well as "a good emulsifier" (sorbitan oleate type) to obtain a good stable emulsion. As is discussed above, the situation is different for emulsion/nitrate blends. The optimum cell size of the internal phase of an emulsion in a blend is the largest that will not crystallize on losing water over the goal shelf life of the product. This insures a minimum rate of water transfer, without premature crystallization of the emulsion. The optimum cell size generally is from about 1 to about 4 microns, decreasing as the aqueous phase water content decreases.

Detailed Description Text (18):

In the present invention, oils and aqueous inorganic oxidizing salts solutions known to the explosive emulsion art may be employed, preferably those disclosed in the aforementioned U.S. Pat. No. 4,287,010. Most often, the inorganic oxidizing salt present in the emulsion's aqueous phase will be an ammonium, alkali metal, or alkaline earth metal nitrate or perchlorate, preferably ammonium nitrate, alone or in combination with, for example, up to 50 percent sodium nitrate (based on the total weight of inorganic oxidizing salts in the aqueous phase). Salts having monovalent cations are preferred, as explained in U.S. Pat. No. 4,287,010. Suitable oils for use in the liquid carbonaceous fuel include fuel oils and lube oils of heavy aromatic, naphthenic, or paraffinic stock, mineral oil, dewaxed oil, etc.

Detailed Description Text (26):

Explosives which are blends of a water-in-oil emulsion and nitrate, preferably AN or ANFO, prills having a physical and chemical structure that minimizes water loss and transport from the emulsion's aqueous phase according to the method of the invention, and especially blends of the "high oil" emulsion of the invention and nitrate prills, are useful in bulk as well as packaged form. The emulsion/nitrate blend of the invention made with the low-viscosity "high oil" emulsion, and particularly the preferred "in situ" emulsion, is especially suited for pumping operations. A preferred technique for pumping the blend into a borehole is to pump it through an annular stream of aqueous lubricating liquid, e.g., naturally occurring water, flowing through the conduit used to transfer the blend to the hole. Such a technique is described in U.S. Pat. No. 4,462,429, issued July 31, 1984, to D. L. Coursen, for pumping a Bingham solid, e.g., a water-in-oil emulsion explosive. By use of a method and apparatus of the type described in the Coursen patent, the disclosure of which is incorporated herein by reference, the resistance of the emulsion/nitrate blend to movement through a conduit is reduced by provision of an annular layer of liquid of low viscosity, e.g., water, around a central column of the blend in the conduit. An annulus of aqueous lubricating liquid, injected into the conduit through which the emulsion/nitrate blend is to be delivered to the borehole, provides lubrication sufficient to permit a column of the blend to slide through the conduit without undergoing appreciable deformation in shear, i.e., movement in "plug flow", a distinct benefit for maintaining the emulsion structure of the blend. An additional benefit of using this apparatus is that it is more effective when used with small amounts of lubricant, which assures better control of the strength and sensitivity of the explosive blend owing to the decreased risk of dilution. A lubricating liquid flow rate which is no greater than about 5%, and usually no greater than about 0.5-2%, of the emulsion/nitrate blend

flow rate is used.

Detailed Description Text (28):

The above-described annular lubricant method can be carried out with intermittent pumping, if desired, even in the case in which water is the lubricating liquid. In contrast to the process described in U.S. Pat. No. 4,259,977 for pumping emulsions, in the present process, in which the material being pumped is an emulsion laden with solid nitrate, plugging of the delivery conduit does not occur on stoppage of the pumping operation when a water annulus is used. It is believed that the avoidance of the swelling/plugging problem in the annular lubricant pumping method is related to the nature of the continuous phase in the explosive emulsion used in the present blend, and more particularly to the hydrophobicity thereof resulting from the emulsifying agent or system therein. It is possible that the fatty acid salt, and especially the equilibrium structure of the emulsifying system produced when the emulsifying agent is formed in situ, as is described in the aforementioned U.S. Pat. No. 4,287,010, provide a uniquely hydrophobic environment between the lubricating liquid on the outer surface of the emulsion/nitrate blend and the aqueous phase droplets within the blend, thereby preventing the absorption of the lubricating liquid into the blend despite the presence of a concentration gradient between the lubricating liquid and the aqueous phase droplets. In any event, a matching of such concentrations is unnecessary with the present blends, and any available water supply can be used to provide the lubricating liquid.

CLAIMS:

27. A water-in-oil emulsion adapted to be blended with inorganic nitrate prills to form an explosive, said emulsion comprising

(a) about from 7 to 21 percent by weight of a liquid carbonaceous fuel including an oil solution of a fatty acid, said solution forming a continuous emulsion phase;

(b) an aqueous solution of an inorganic oxidizing salt forming a discontinuous emulsion phase dispersed as discrete droplets within said continuous phase; and

(c) an emulsifying system comprising (1) said fatty acid and (2) a fatty acid salt, said oil, fatty acid, and fatty acid salt together forming said liquid carbonaceous fuel, and the ratio of the amounts of oil and fatty acid added to form said emulsion being in the range of about from 1/1 to 3/1 by weight; said emulsion having an oxygen balance more negative than about -6 percent.

35. An essentially oxygen-balanced water-in-oil emulsion adapted to be blended with inorganic nitrate prills and oil to form an explosive, said emulsion comprising

(a) a liquid carbonaceous fuel including an oil solution of a fatty acid, said solution forming a continuous emulsion phase;

(b) an aqueous solution of an inorganic oxidizing salt forming a discontinuous emulsion phase dispersed as discrete droplets within said continuous phase; and

(c) an emulsifying system comprising (1) said fatty acid and (2) a fatty acid salt, said oil, fatty acid, and fatty acid salt together forming said liquid carbonaceous fuel;

said emulsion being devoid of a sensitizing amount of dispersed gas bubbles or voids.

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File: USPT

Nov 2, 1982

DOCUMENT-IDENTIFIER: US 4357184 A

TITLE: Explosive compositions based on time-stable colloidal dispersions

Brief Summary Text (10):

The water-in-oil microemulsion explosive compositions of the invention comprise essentially an aqueous solution of at least one oxygen-supplying salt as a discontinuous phase, an insoluble liquid or liquefiable carbonaceous fuel as a continuous phase, at least one sensitizing component distributed substantially homogeneously throughout the composition as a further discontinuous phase and a distinct definable blend of emulsifying agents capable of producing a time-stable microemulsion. The compositions may optionally contain particulate oxygen-supplying salts, ANFO, particulate light metals, particulate fuels, particulate solid explosives, soluble and partly soluble self-explosives, explosive oils and the like for purposes of augmenting the strength and sensitivity or decreasing the cost of the compositions. The specific blends of emulsifiers capable of producing a time-stable, water-in-oil microemulsion explosive composition comprise a mixture of at least one amphiphatic synthetic polymeric emulsifier selected from graft, block or branch polymers and at least one conventional water-in-oil emulsifier. Optionally a phosphatide emulsion stabilizer may be included in the blend. By "amphiphatic graft, block or branch polymers" is meant a polymer comprising at least two or more segments, one of which is only soluble in an oil phase and the other only soluble in an aqueous phase, each segment having a molecular weight of at least 500. By "conventional water-in-oil emulsifier" is meant herein the relatively low molecular weight emulsifiers which are capable of producing a water-in-oil emulsion. Most of these emulsifiers are listed in the well known publication "McCutcheon's Detergents & Emulsifiers".

Brief Summary Text (53):

The sensitizing component distributed substantially homogeneously throughout the composition is preferably occluded gas bubbles which may be introduced in the form of glass or resin microspheres or other gas-containing particulate materials. Alternatively, gas bubbles may be generated in-situ by adding to the composition and distributing therein a gas-generating material such as, for example, an aqueous solution of sodium nitrite. Other suitable sensitizing components which may be employed alone or in addition to the occluded or in-situ generated gas bubbles include insoluble particulate solid self-explosives such as, for example, grained or flaked TNT, DNT, RDX and the like and water soluble and/or hydrocarbon soluble organic sensitizers such as, for example, amine nitrates, alkanolamine nitrates, hydroxyalkyl nitrates, and the like. The explosive compositions of the present invention may be formulated for a wide range of applications. Any combination of sensitizing components may be selected in order to provide an explosive composition of virtually any desired density, weight-strength or critical diameter.

Brief Summary Text (55):

Optional additional materials may be incorporated in the composition of the invention in order to further improve sensitivity, density, strength, rheology and cost of the final explosive. Typical of materials found useful as optional additives include, for example, emulsion promotion agents such as highly

chlorinated paraffinic hydrocarbons, particulate oxygen-supplying salts such as prilled ammonium nitrate, calcium nitrate, perchlorates, and the like, ammonium nitrate/fuel oil mixtures (ANFO), particulate metal fuels such as aluminium, silicon and the like, particulate non-metal fuels such as sulphur, gilsonite and the like, particulate inert materials such as sodium chloride, barium sulphate and the like, water phase or hydrocarbon phase thickeners such as guar gum, polyacrylamide, carboxymethyl or ethyl cellulose, biopolymers, starches, elastomeric materials, and the like, crosslinkers for the thickeners such as potassium pyroantimonate and the like, buffers or pH controllers such as sodium borate, zinc nitrate and the like, crystals habit modifiers such as alkyl naphthalene sodium sulphonate and the like, liquid phase extenders such as formamide, ethylene glycol and the like and bulking agents and additives of common use in the explosives art.

Brief Summary Text (59):

Characteristic of the novel explosive compositions of the invention is the unique nature of the water-in-oil microemulsion which results from the use of specific blends of emulsifiers. The microemulsion of the invention is a demonstrably different state of matter than any of previously disclosed, conventional prior art explosive emulsions. Several techniques well known to those experienced in the art, may be employed to differentiate the microemulsions of this invention from the conventional explosive emulsions of the prior art.

Brief Summary Text (65):

All prior art explosive emulsions show gradually increasing crystal growth and structure upon storage as a consequence of their instability and slow coalescence of the aqueous oxidizer salt droplets. This increasing crystal structure can be easily detected by X-ray diffraction. The microemulsion explosives of this invention show no such X-ray diffraction pattern even at very low temperature or after prolonged storage and/or for compositions containing extremely low levels of water.

Detailed Description Text (16):

A further aspect of the microemulsion explosive composition of the present invention is that doping with substantially large proportions of, for example, energy enhancing solid materials such as solid AN prills, does not significantly alter the sensitivity or the stability of the composition. Furthermore, if the microemulsion composition is formulated so as to possess a suitably high fluidity, a very large proportion of these solid materials may be added without significant loss of pumpability. Retention of fluidity is not usually the case with water-gel explosives; the addition of extra amounts of high energy content ingredients such as AN prills is severely restricted because of rapid loss of pumpability, reduction in initiator sensitivity levels and in water resistance qualities. Doping of the microemulsion compositions of this invention at levels up to 50% addition of extra AN prills or ANFO, nevertheless produces waterproof pumpable explosive compositions which retain booster sensitivity at densities up to 1.35 g/cc. These new explosive compositions have weight strengths which are substantially higher than the weight strength of the undoped microemulsion and, more importantly, the raw material costs are at the same time substantially reduced. Furthermore, the blasting performance which is in part determined by calculable gas evolution volumes, is substantially higher than that of other pumpable, waterproof explosive compositions because of the much higher proportion of gas producing AN contained in these new explosive compositions. In addition, the other unique explosive properties associated with the microemulsion compositions such as high velocities of detonation, very small critical diameters, temperature independent sensitivity, and storage stability, are substantially retained by the doped microemulsion compositions.

CLAIMS:

15. A water-in-oil microemulsion explosive composition as claimed in claim 1

wherein the water content comprises 3.0% to 30.0% by weight of the total composition.

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Search Results -

Terms	Documents
L5 and strength	13

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<u>L4</u>	(water adj content)	57065	<u>L4</u>
<u>L3</u>	L1 and 102.clas.	3	<u>L3</u>
<u>L2</u>	L1 and 149.clas.	4	<u>L2</u>
<u>L1</u>	strength same (water adj content)	3613	<u>L1</u>

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